IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Roland Deckwer et al.

Application No.: 10/734,828 Confirmation No.: 5323

Filed: December 12, 2003 Art Unit: 1616

For: OIL SUSPENSION CONCENTRATE Examiner: A. N. Pryor

DECLARATION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Dr. Roland Deckwer, state that I reside at Königsteinerstrasse 92a, D-65929 Frankfurt/Main; I am a citizen of the Federal Republic of Germany; that I am familiar with the subject matter and the prosecution of the instant application Serial No. 10/734,828 filed December 12, 2003, entitled "Oil Suspension Concentrate"; that I consider myself qualified by my education, knowledge and experience in physical chemistry and formulation technology to make this Declaration; and that I have made the following observations:

This declaration refers to my previous declaration, especially to Table 3 herein (see Enclosure). The statement that "all oil suspension concentrates summarized in Table 3, show good chemical and physical stability while storage (8w40°C) ... " can be substantiated by the following results:

- chemical stable means that the relative degradation of the active ingredient after storage (8w40°C) is below 10%, measured by HPLC (High Performance Liquid Chromatography)

- physical stable means that after storage (8w40°C) the average particle size stays below 8µm (d50), measured by microscopy (standard method by microscope manufacturer, e.g. Leica, USA, Model Galen III)

- physical stable means that after storage (8w40°C) no sediment is formed in the formulation, measured by standard method (visual control)

- physical stable means that after storage (8w40°C) the wet sieve residues of the formulation retaining on a 75µm test sieve are below 0.1%, following CIPAC method MT185, CIPAC Handbook K, ISBN 0902951157

- physical stable means that after storage (8w40°C) the formulation shows in the dispersion stability test less than 2% of cream, only traces of oil and no sediment after 30min of standing and that the dispersion is re-homogenizable after 24 hours of standing, following CIPAC method MT180, CIPAC HANDBOOK H, ISBN 0902951130, MT180 is written for the dispersion stability test of suspo-emulsions, but could be assigned to oil dispersions and is already a required part of the registration dossier in many countries, a special method for the dispersion stability test of oil dispersions is under preparation by CIPAC

  All abbreviations used here are explained in my previous declaration. For the description of the CIPAC methods see Enclosure.

These results are effective for all herbicidal active ingredients summarized in Table 3 which are wide choice examples of the group of sulfonamides.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing there from.

Signed: Roland Deckwer

Name: Dr. Roland Deckwer

Date: 09.02.2010

Enclosures

ENCLOSURE

Example 3/ Example 4 – Table 3
Variation of components

Example	3.2 in %w/w	4.1 in %w/w	4.2 in %w/w	4.3 in %w/w	4.4 in %w/w	4.5 in %w/w	4.6 in %w/w	4.7 in %w/w	4.8 in %w/w	4.9 in %w/w
Foramsulfuron (acid)		3.00								
Mesosulfuron			4.50							
Ethoxysulfuron				2.50						
Amidosulfuron					10.00					
Propoxycarbazon						3.00				
Flucarbazon							3.00			
A21.1								1.00	1.00	1.00
Thifensulfuron	8.00									
Mefenpyr	24.00		12.00		20.00	9.00	10.00	5.00		
Isoxadifen		3.00		9.00					2.00	
Cyprosulfonamid										0.50
Triton ®GR-7M E	25.00	10.00	10.00	12.50	20.00	15.00	15.00	25.00	20.00	20.00
Solvesso® 200		66.50	46.30	58.00	37.50		48.40	50.00	58.00	64.20
Edenor® MESU	32.00		10.00			10.00	10.00			
Bayol® 85						46.60				
Jeffsol® PC						0.20	0.50	1.00	1.00	1.00
Genapol® PF10	5.00				2.50			2.00	2.50	
Emulsogen® EL-400	5.00	3.00						4.00		
Emulsogen® EL-360			3.00	3.00	2.00				3.50	3.00
Genapol® V4739		10.00		10.00		15.00		8.00	8.00	
Genapol® XM150			10.00		8.00		12.00			
Genapol® X060								2.00	2.00	8.00
Emcol® P1860		2.00	2.00	2.50						
Thixatrol® ST	1.00									
Bentone® SD-1			2.20					2.00		
Bentone® 38		2.50		2.50					2.00	2.30
Bentone® 34						1.20	1.10			

ENCLOSURE

MISCELLANEOUS TECHNIQUES

*MT 180 DISPERSION STABILITY OF SUSPO-EMULSIONS

SCOPE AND LIMITATIONS

The method is suitable for the determination of the dispersion stability of suspo-emulsions (SE) (Note 1).

OUTLINE OF METHOD

A dispersion of prescribed concentration in Standard Water is prepared and aliquots are placed in two graduated emulsion tubes, which are then allowed to remain undisturbed for specified time in a upright and inverted positions at a constant temperature. The dispersion characteristics are observed immediately after the preparation of the dispersion, after a specified time, and after re-dispersion.

REAGENT

Standard Water MT 18

APPARATUS

Emulsion tubes ASTM centrifuge tubes, borosilicate glass with conical bottom, 15 cm (6 inch), graduated to 100 ml (ASTM D 91 and D 96)
Rubber stopper fitting in the emulsion tube and provided with a 80 mm glass ventilation tube (external diameter: 4.5 mm; internal diameter: 2.5 mm, see Fig. 27).
Graduated cylinders 250 ml, as for MT 15 (CIPAC F, *p.*46)
Adjustable lamp fitted with a 60 W pearl bulb (Angle Poise or equivalent)
Pipette 10 ml

PROCEDURE

Fill at room temperature (23 ± 2 °C, Note 2) two graduated cylinders (250 ml) to the 240 ml mark with Standard Water, to each add dropwise by pipette 5 g of sample (or such an amount as otherwise is specified). Dispense the sample with the tip of the pipette as closely as possible to the surface of the water but not beneath it. Fill to the mark with Standard Water (Note 3).

*Provisional CIPAC method 1997

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Invert the cylinder 30 times by holding the cylinder with one hand at each end, insulated with a cloth, and rotating it through 180 degrees and back again through an imaginary fixed point midway between the hands. Ensure that no 'bouncing' occurs. Each inversion should take 2 seconds (observe a stopwatch!). Use the content of one cylinder for the sedimentation and creaming tests *(ii)* and *(iii)* respectively and keep the second one for the determination of the re-dispersion test *(iv)*.

(i)Initial determination
Observe the dispersions and note any sediment or cream or oil.

(ii) Determination of sediment volume
Immediately after forming of the dispersion transfer a 100 ml aliquot from the first graduated cylinder to an emulsion tube. Stopper and allow to stand in an upright position at room temperature (Note 2) for 30 min. Illuminate the cylinder with the lamp. Adjust the position and the angle of the light for optimal viewing of the phase boundary, if present (it is usually easier to see it by reflected, rather than by transmitted light). Record the sediment volume with an accuracy of ± 0.05 ml.

(iii) Determination of top cream (or oil) volume
Immediately after forming of the dispersion fill an emulsion tube to within about 1 mm from the top of the tube with the dispersion. Wearing disposable protection gloves stopper the tube with the rubber stopper (fitted with a ventilation tube) in such a way that all air is expelled from the tube. Carefully remove from the apparatus any expelled dispersion. Invert the tube and maintain it in an upside down position at room temperature for 30 min. No liquid will escape from the emulsion tube and it is not necessary to seal the open end of the glass tube. Record the volume of cream or oil that has formed. Determine the total volume of the tube and correct the measured volume of cream or oil in the following way:

$$F = \frac{100}{V_0}$$

where:
F = correction factor to be applied to the measured volume of cream or oil
V_0 = total volume of the emulsion tube

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(iv) Determination of re-dispersion
After the initial dispersion, allow the second cylinder to stand undisturbed for 24 h at room temperature. To re-disperse the contents invert the cylinder 30 times through 180° and back, as described above. Note any bottom sediment that is not re-dispersed completely. Add the dispersion to fresh emulsion tubes as described under *(ii)* and *(iii)* and measure the sediment volume or top cream or oil volume of the re-dispersed product after standing for 30 min.

RESULTS

Report the results of the dispersibility test as follows:

Initial determination (after standing for 30 min)
sediment volume..... ml
top cream (or oil)..... ml

Re-dispersibility (after standing for 24 h)
sediment volume..... ml
top cream (or oil)..... ml

Note whether the bottom sediment was completely re-dispersed.

Note 1 The method can be used as screening method for the dispersion characteristics of other water dispersible formulations such as wettable powders, water dispersible granules, suspension concentrates, emulsifiable concentrates, and oil in water emulsions.

Note 2 Report the temperature and range if they are outside the range indicated.

Note 3 In the case of solid formulations shake 5 g of powder carefully onto the surface of the water.

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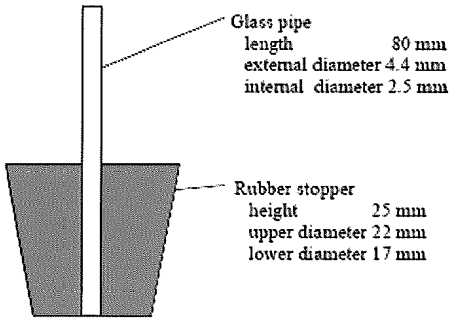

Fig. 26 Rubber stopper with glass ventilation tube

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MISCELLANEAOUS TECHNIQUES

*MT 185 WET SIEVE TEST

SCOPE The method is suitable for the determination of the amount of non-dispersible material in formulations, that are applied as dispersions in water.

OUTLINE OF METHOD A sample of the formulation is dispersed in water and the suspension formed is transferred to a sieve and washed. The amount of the material retained on the sieve is determined by drying and weighing.

APPARATUS

Balance with an accuracy of at least ± 0.01g
Beaker 250 ml
Magnetic stirrer and magnetic flea (stirring bar)
Rubber hose of approx. 10 mm internal diameter
Oven
Desiccator
Sieve 20 cm diameter, 75 µm mesh size (200 mesh according to ASTM E 11-61, 0.075 mm according to ISO 565, DIN 4188), if not otherwise specified

PROCEDURE

(a) Wetting
Weigh (to the nearest 0.1 g) 10 g (or if necessary a more appropriate quantity) of the sample into a beaker (250 ml) and add tap water (100 ml) (Note 1). Allow to stand for 60 s. Then stir with the magnetic stirrer for 5 min, making no deliberate attempt to break up any lumps (Note 2).

(b) Wet sieving Transfer the slurry to the sieve, rinsing with tap water, removing the magnetic flea (having washed any dispersed material from the flea into the sieve). Wash the material on the sieve with a jet of tap water (Note 3) using a rubber hose of 10 mm i.d. delivering 4-5 litres of water per min. Continue the washing until the visible quantity of residue remains constant (max. 10 min). Direct the water from the circumference of the sieve towards the centre and keep the end of the hose at a distance of between 2 - 5 cm from the surface of the sieve (Note 4). Transfer the residue to a tared glass dish with a jet of deionised water from a wash bottle. Dry to constant weight (Note 5) and record the weight of the sample to the nearest 0.01 g.

* CIPAC method 20001. Prepared by the German Formulation Panel (DAPF). Chairman: G Menschel.

ENCLOSURE

MISCELLANEAOUS TECHNIQUES

CALCULATION

Express the weight of residue as a percentage of the sample weight and record the result as a percentage retention on the stated test sieve.

Note 1 This method was developed using tap water of temperature from 5 – 15 °C.

Note 2 The speed of rotation of the magnetic flea should be chosen such that a vortex just forms on the surface of the liquid. The shear forces generated this way approach those found in commercial spray equipment. Care should be taken that the dispersion does not become aerated by over-vigorous dispersion of the sample.

Note 3 The tap water may contain solids, and should, if required, be screened before use.

Note 4 The washing water may be recycled along the lines described in CIPAC MT 182.

Note 5 A temperature of 60-70°C is recommended. If necessary, the temperature must be varied to avoid decomposition or volatilisation at the drying temperature.